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QUINOXALINE-BENZIMIDAZOLE REARRANGEMENTS IN THE REACTIONS OF 3-ALKANOYLQUINOXALIN-2-ONES WITH 1,2-PHENYLENEDIAMINES

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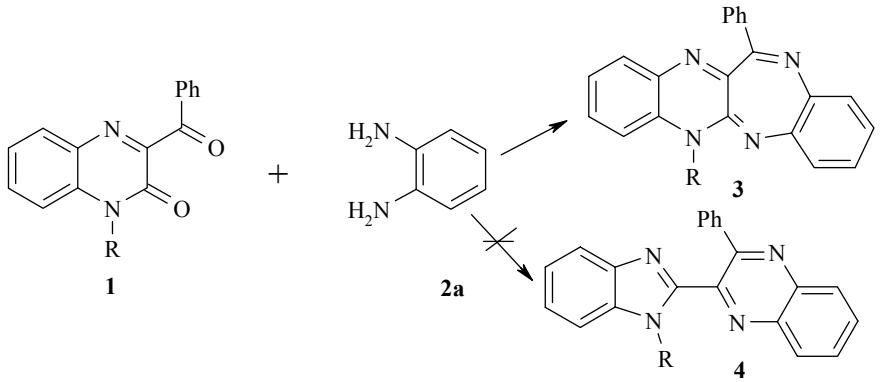
The interaction of 3-alkanoylquinoxalin-2-ones with 1,2-phenylenediamines in boiling acetic acid led to the contraction of the pyrazine ring as the result of a quinoxaline-benzimidiazole rearrangement with the formation of 2-benzimidazolyl-substituted quinoxalines.

Keywords: 3-alkanoylquinoxalin-2-ones, benzimidazole, 1,2-phenylenediamines, IR and ^{1}H NMR spectra, rearrangements, ring contraction.

The benzimidazole system is a structural unit of vitamin B_{12} [1, 2] and some derivatives of benzimidazole are made industrially as medicinal preparations [3], substances used in veterinary [4], and fungicides [5]. The most widely used methods for the synthesis of benzimidazole system include the introduction of atom C(2) between the nitrogen atoms in various *ortho*-di-N-substituted derivatives of benzene [6-10] and intramolecular substitution of N-phenylamidines [11,12], or their relative compounds [13-15]. Apart from the Phillips-Ladenburg reaction [16, 17] there is no general method for the introduction of the benzimidazole unit into different heterocyclic systems.

We found previously that the reaction of 3-benzoylquinoxalin-2-ones **1** with *o*-phenylenediamine **2** in boiling acetic acid occurs with the formation of products of a quinoxaline-benzimidazole rearrangement – 2-(benzimidazol-2-yl)-3-phenylquinoxalines **4** [18, 19] – in place of the expected quinoxalino[2,3-*b*]benzo-1,5-diazepines **3** (Scheme 1):



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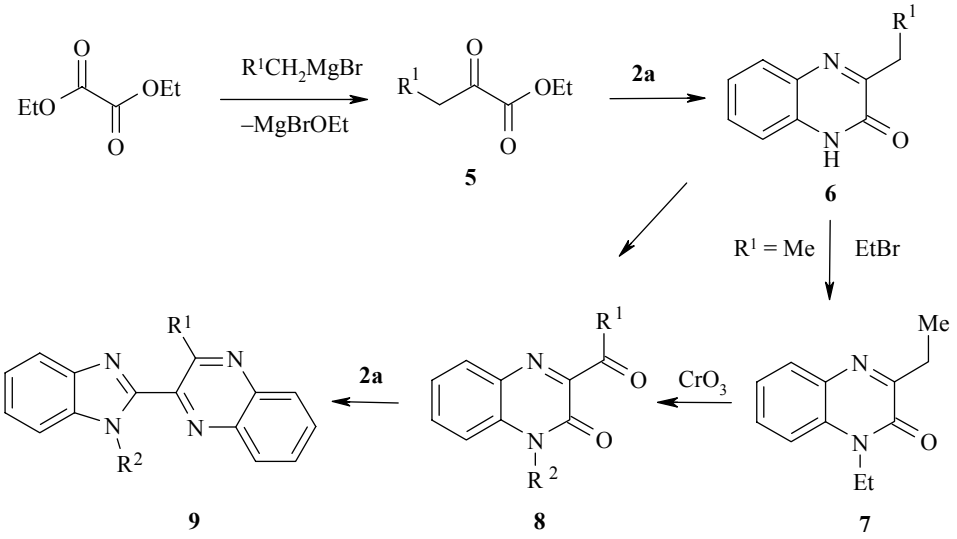
In the present work we have extended this rearrangement to 3-alkanoylquinoxalinones.

In Scheme 2 the synthesis of 3-alkanoylquinoxalin-2-ones is shown based on the esters of α-keto acids **5** – the products of the reaction of diethyl oxalate with alkylmagnesium bromides. The esters were converted into 3-alkylquinoxalin-2-ones **6** and **7** [20], the oxidation of which with CrO_3 gave the alkanoylquinoxalinone **8**. Condensation of compounds **8** with *o*-phenylenediamine **2a** by a method which we developed previously [19] (boiling for 1 h in acetic acid) led to the formation of crystalline 3-alkyl-2-benzimidazoylquinoxalines **9**, alkyl analogs of compound **4.** The absence of oxygen in their composition was confirmed by elemental analysis and the absence of carbonyl absorptions in their IR spectra.

Scheme 2



5, **6 a** R^1 = Me, **b** R^1 = Pr; **8**, **9 a** R^1 = Me, R^2 = H, **b** R^1 = Pr, R^2 = H, **c** R^1 = Me, R^2 = Et

The fact that attracts the attention is the shift by about 0.7 ppm to weak field of the singlet signal of the methyl (compounds **9a,c**) and the quartet signal of the methylene groups (compound **9b**) in the ^{1}H NMR spectra of the products in comparison with the proton signals in the spectra of alkanoylquinoxalin-2-one starting materials (**8a**,**c**), which indicates a rearrangement with the shift of these substituents from the alkanoyl groups to the directly adjacent heteroaromatic systems, in which, thanks to the "ring current effect", an additional descreening of these hydrogen atoms is observed [21].

As a result of this reaction the carbon fragment Alk–C(O)–C(3)–C(2) of the initial alkanoylquinoxalinones **8** is transferred completely into compounds **9**, where it is found in the structure of the alkyl-substituted quinoxaline ring of the product as the Alk–C(3)–C(2) and the newly constructed benzimidazole ring as the μ-carbon atom. Such a reaction falls under the most general definition of a molecular rearrangement as a chemical reaction with the change in the molecular skeleton with the violation of the principle of minimal structural changes [22].

This rearrangement is extended to the podands terminal 3-acetylquinoxalinone units. For example, 1,5-bis(3-acetyl)-2-oxoquinoxalin-1-yl)-3-oxapentane (**11**) [23] easily obtained by alkylation of 3-ethylquinoxalin-3-one **6** [20] with bis(2-bromoethyl) ether and by oxidation of the obtained product **10** with CrO_3, reacted with *o*-phenylenediamine to give in high yield 1,5-bis[2-(3-methylquinoxalin-2-yl)benzimidazol-1-yl]-3-oxapentane (**12**) (Scheme 3).

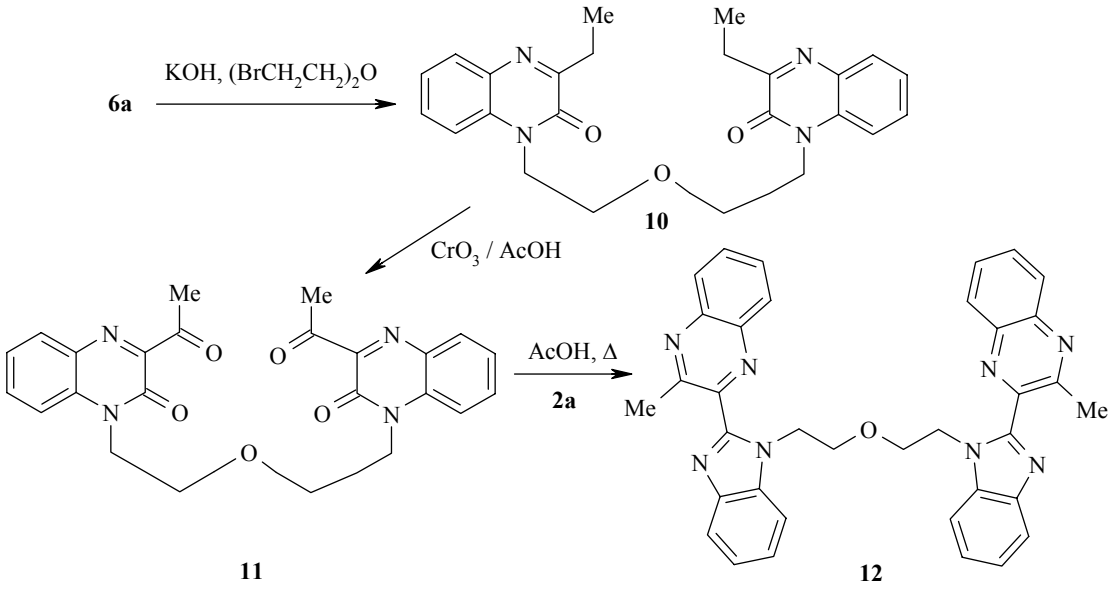
Table 1. Characteristics of the Synthesized Compounds **6-14**

Compound	Empirical formula	Found, % / Calculated, %			mp, °C*	Yield, %
		C	H	N		
6b	$C_{12}H_{14}N_2O$	71.34 / 71.26	7.09 / 6.98	14.04 / 13.85	258-260	78
8b	$C_{12}H_{12}N_2O_2$	66.78 / 66.65	5.73 / 5.59	12.89 / 12.95	165-166	28
9a	$C_{16}H_{12}N_4$	73.90 / 73.83	4.52 / 4.64	21.46 / 21.52	176-178	72
9b	$C_{18}H_{16}N_4$	74.99 / 74.97	5.43 / 5.59	19.38 / 19.43	186-188	75
9c	$C_{18}H_{16}N_4$	74.82 / 74.97	5.47 / 5.59	19.57 / 19.43	125-127	99
10	$C_{24}H_{26}N_4O_3$	68.73 / 68.88	6.35 / 6.26	13.24 / 13.39	164-166	89
11	$C_{24}H_{22}N_4O_5$	64.67 / 64.57	5.04 / 4.97	12.42 / 12.55	162-164	60
12	$C_{36}H_{30}N_8O$	73.35 / 73.20	5.02 / 5.12	19.07 / 18.97	189-191	75
13a + **14a**	$C_{16}H_{11}N_5O_2$	63.06 / 62.95	3.57 / 3.63	23.08 / 22.94	299-339	52
13b + **14b**	$C_{17}H_{14}N_4$	73.34 / 73.43	5.23 / 5.14	20.56 / 20.42	203-205	72

* Solvents: *i*-PrOH (compounds **6b**,**9a**), MeOH (compound **9b**), EtOH (compound **9c**), acetone (compounds **10**-**12**).

All of the compounds in the rearrangement sequence **6a** → **10** → **11**→ **12** were obtained in high yield. A characteristic signs which indicated the formation of these products in these reactions, in the first step is the presence in their ^{1}H NMR spectra, along with other signals, of two triplets signals of the fragment $CH_2CH_2OCH_2CH_2$ at δ 3.82 and 4.39 ppm and the presence in the IR spectra of an absorption of the carbamoyl carbonyl group ($\nu_{C=O}$ = 1649 cm^{-1}), and in the second step the disappearance from the ^{1}H NMR spectra of the triplet and quartet signals of the protons of the ethyl group at δ 1.32 and 2.95 ppm, the appearance of a singlet

Scheme 3



signal in the 2.70 ppm region of the acetyl group and the appearance in the IR spectra of a band in the region of 1714 cm^{-1} ($\nu_{C=O}$), and in the third stage the shift in the ^1H NMR spectrum of the singlet signal of the methyl group and the triplet signal of the N-methylene group in the 3-oxapentane unit to weaker field in comparison with the starting compound **11** (Table 2) and the disappearance in the IR spectrum of the absorption band of the ketone and carbamoyl groups ($\nu_{(C=O)}$).

Table 2. Spectral Characteristics of the Quinoxalines **6-14**

Com-pound	IR spectrum, ν, cm^{-1}	^1H NMR spectrum, δ, ppm (SSCC, *J*, Hz)*
1	2	3
6b	469, 590, 624, 706, 751, 893, 943, 1150, 1287, 1565, 1608, 1666, 2500-3300	0.93 (3H, t, *J* = 7.28, CH_3); 1.33-1.45 (2H, m, $CH_3\underline{CH_2}$); 1.63-1.77 (2H, m, $CH_3CH_2\underline{CH_2}$); 2.80 (2H, t, *J* = 7.28, C(3)CH_2); 7.24 (1H, dd, *J* = 8.28, *J* = 6.96, H-6 and H-7); 7.28 (1H, d, *J* = 7.64, H-8); 7.45 (1H, ddd, *J* = 7. 96, *J* = 7.28, *J* = 1.00, H-6 and H-7); 7.69 (1H, d, *J* = 8.28, H-5); 12.08 (1H, br. s, NH)
8b	416, 475, 542, 555, 593, 728, 768, 792, 832, 892, 968, 1140, 1179, 1264, 1300, 1336, 1400, 1421, 1464, 1490, 1611, 1643, 1710, 2500-3200	1.05 (3H, t, *J* = 7.54, CH_3); 1.77-1.85 (2H, m, $CH_3\underline{CH_2}$); 3.15 (2H, t, *J* = 7.20, CH_2CO); 7.42 (1H, dd, *J* = 7.55, *J* = 7.54, H-6 and H-7); 7.47 (1H, d, *J* = 7.89, H-8); 7.64 (1H, dd, *J* = 7.55, *J* = 6.85, H-6 and H-7); 7.93 (1H, d, *J* = 7.54, H-5); 12.73 (1H, br. s, NH)
9a	430, 738, 766, 955, 1010, 1073, 1133, 1180, 1214, 1253, 1274, 1320, 1338, 1486, 2600-3400	3.43 (3H, s, CH_3); 7.33-7.40 (2H, m, H-5,6 benzimidazole); 7.70-7.80 (4H, m, H-4,7 benzimidazole , H-6,7 quinoxaline); 8.05-8.11 (2H, m, H-5,8 quinoxaline)
9b	433, 578, 608, 729, 746, 764, 954, 1063, 1089, 1145, 1172, 1209, 1277, 1317, 1344, 1422, 1485, 1549, 3397	1.06 (3H, t, *J* = 7.56, CH_3); 1.80-2.00 (2H, m, $CH_3\underline{CH_2}$); 3.80 (2H, t, *J* = 7.56, C(3)CH_2); 7.28-7.38 (2H, m, benzimidazole); 7.70-7.80 (2H, m, benzimidazole); 7.89-7.93 (2H, m, quinoxaline); 8.10-8.15 (1H, m, quinoxaline); 8.16-8.25 (1H, m, quinoxaline)
9c	433, 738, 754, 767, 1008, 1038, 1123, 1138, 1174, 1209, 1289, 1309, 1334, 1376, 1412, 1451, 1484, 1559, 1611	1.53 (3H, t, *J* = 7.28, $CH_2\underline{CH_3}$); 2.97 (2H, s, C(3)CH_3); 4.49 (2H, q, *J* = 7.28, $\underline{CH_2}CH_3$); 7.36 (1H, ddd, *J* = 7.32, *J* = 6.88, *J* = 0.88, H-5 and H-6 benzimidazole); 7.43 (1H, ddd, *J* = 8.16, *J* = 7.32, *J* = 0.84, H-5 and H-6 benzimidazole); 7.67 (1H, d, *J* = 8.16, H-4 and H-7 benzimidazole); 7.81 (1H, d, *J* = 8.16, H-4 and H-7 benzimidazole); 7.82–7.91 (2H, m, H-6 and H-7 quinoxaline); 8.09 (1H, dd, *J* = 8.56, *J* = 1.68, H-5 and H-8 quinoxaline); 8.14 (1H, dd, *J* = 7.72, *J* = 1.72, H-5 and H-8 quinoxaline)
10	442, 460, 522, 562, 581, 635, 717, 748, 888, 940, 954, 986, 1049, 1075, 1192, 1114, 1177, 1224, 1262, 1313, 1353, 1371, 1424, 1467, 1487, 1569, 1604, 1649	1.32 (6H, t, *J* = 7.30, CH_3); 2.95 (4H, q, *J* = 7.30, $\underline{CH_2}CH_3$); 3.82 (4H, t, *J* = 5.60, OCH_2); 4.39 (4H, t, *J* = 5. 60, NCH_2,); 7.12-7.49 (6H, m, H-6–8 quinoxaline); 7.82 (2H, d, *J* = 8.15, H-5 quinoxaline)
11	462, 513, 542, 556, 594, 625, 661, 718, 762, 859, 888, 943, 1008, 1038, 1083, 1111, 1127, 1142, 1162, 1201, 1240, 1276, 1317, 1340, 1539, 1583, 1603, 1649, 1714	2.70 (6H, s, CH_3); 3.84 (4H, t, *J* = 5.63, OCH_2); 4.41 (4H, t, *J* = 5.63, NCH_2); 7.32 (2H, d, *J* = 7.30, H-6 and H-7 quinoxaline); 7.37 (2H, d, *J* = 7.80, H-8 quinoxaline); 7.53 (2H, ddd, *J* = 7.80, *J* = 7.15, *J* = 1.30, H-6 and H-7 quinoxaline); 7.91 (2H, dd, *J* = 8.20, *J* = 1.30, H-5 quinoxaline)

Table 2. (continued)

1	2	3
12	432, 736, 763, 1006, 1033, 1120, 1165, 1308, 1334, 1407, 1482, 1561, 1610	2.84 (6H, s, CH_3); 3.69 (4H, t, *J* = 5.54, OCH_2); 4.50 (4H, t, *J* = 5.54, NCH_2); 7.22 (2H, dd, *J* = 7.20, *J* = 7.16, H-5 and H-6 benzimidazole); 7.29 (2H, dd, *J* = 7.64, *J* = 7.16, H-5 and H-6 benzimidazole); 7.46 (2H, d, *J* = 8.04, H-4 and H-7 benzimidazole); 7.77 (2H, d, *J* = 8.04, H-4 and H-7 benzimidazole); 7.82 (2H, ddd, *J* = 7.62, *J* = 7.48, *J* = 1.48, H-6 and H-7 quinoxaline); 7.89 (2H, ddd, *J* = 7. 62, *J* = 7.62, *J* = 1.48, H-6 and H-7 quinoxaline); 7.98 (2H, d, *J* = 8.36, H-5 and H-8 quinoxaline); 8.06 (2H, d, *J* = 8.36, H-5 and H-8 quinoxaline)
13a + **+14a**	430, 587, 623, 652, 693, 723, 740, 788, 900, 935, 968, 1016, 1068, 1144, 1193, 1234, 1345, 1514, 1557, 1581, 1616	3.29 (3H, s, $CH_3C(3)$ compound **13a** and $CH_3C(3)$ compound. **14a**); 3.295 (3H, s, $CH_3C(3)$ compound **13a** and $CH_3C(3)$ compound. **14a**); 7.25-7.40 (4H, m, H-5,6 benzimidazole); 7.70-7.80 (4H, m, H-4,7 benzimidazole); 8.29 (1H, d, *J* = 9.28, H-8 compound. **13a** and H-5 compound **14a**); 8.34 (1H, d, *J* = 9.28, H-8 compound **13a** and H-5 compound **14a**); 8.55 (2H, dd, *J* = 9.28, *J* = 2.32, H-7, compound **13a** and H-6 compound **14a**); 8.82 (1H, d, *J* = 2.32, H-5 compound **13a** and H-8 compound **14a**); 8.89 (1H, d, *J* = 2.32, H-5 compound **13a** and H-8 compound **14a**); 12.22 (2H, br. s, NH)
13b+ **+14b**	432, 654, 721, 741, 791, 849, 901, 969, 1015, 1070, 1192, 1324, 1347, 1514, 1558, 1616	2.62 (3H, s, $CH_3C(6)$ compound **13b** and $CH_3C(7)$ compound **14b**); 2.63 (3H, s, $CH_3C(6)$ compound **13b** and $CH_3C(7)$ compound **14b**); 3.29 (3H, s, $CH_3C(3)$); 3.295 (3H, s, $CH_3C(3)$); 7.33-7.39 (4H, m, H-5,6 benzimidazole); 7.68 (1H, dd, *J* = 8.40, *J* = 1.56, H-7 compound **13b** and H-6 compound **14b**); 7.69 (1H, dd, *J* = 8.40, *J* = 1.80, H-7 compound **13b** and H-6 compound **14b**); 7.72-7.79 (4H, m, H-4,7 benzimidazole); 7.84 (1H, s, H-5 compound **13b** and H-8 compound **14b**); 7.91 (1H, s, H-5 compound **13b** and H-8 compound **14b**); 7.94 (1H, d, *J* = 8.6, 4H-8, compound **13b** and H-5 compound **14b**); 8.02 (1H, d, *J* = 8.40, H-8 compound **13b** and H-5 compound **14b**)

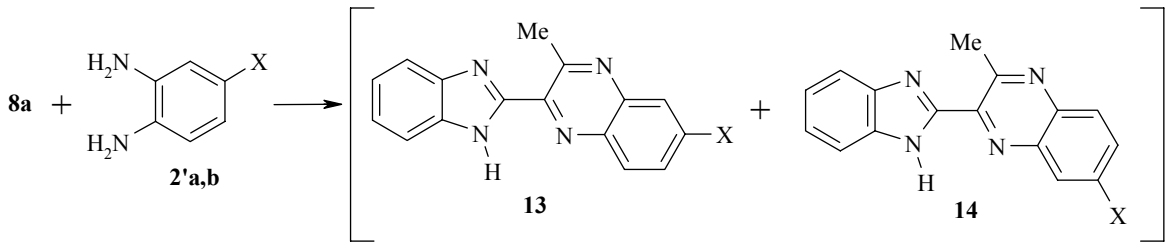
* ^{1}H NMR spectrum in DMSO-d_6 (compounds **6b**, **9b**, **12**, **13a**+**14a**), $CDCl_3$ (compounds **8b**, **9a**, **10**, **11**), and CD_3CN (compounds **9c**, **13b**+**14b**).

A study of the reaction of 3-acetyl-2-oxo-1,2-dihydroquinoxaline **8a** with 1,2-phenylenediamines, which differ markedly in the character of the electronic influence of the substituent in the benzene ring (nitro (**2′a**) and methyl (**2′b)**), showed that, as in the case of 3-benzoyl-2-oxo-1,2-dihydroquinoxalines [19], in these reactions, independent of the nature of the substituents in the benzene rings of the 1,2-phenylenediamines, approximately equal amounts of the isomeric products of the rearrangement **13** and **14** are formed which differ in the substituents in positions 6 and 7 of the quinoxaline system (Scheme 4).

This indicates that either the probability of the attack of the amino group on atom C(3) or on the alkanoyl group during the quinoxaline-benzimidazole rearrangement is approximately equal, or this and subsequent stages are not limiting, or it is not realized either one of these variants of attack in the initial stages of the rearrangement.

So it is found that the interaction of 3-aminoquinoxalin-2-ones and their N'-alkyl derivatives with 1,2-phenylenediamines proceeds *via* quinoxaline-benzimidazole rearrangements to give 2-benzimidazolyl-substituted quinoxalines.

Scheme 4



a X = NO_2, **b** X = Me

EXPERIMENTAL

Melting points were determined on a Boetius block. IR spectra of nujol mulls were recorded with a Bruker Vector-22 Fourier spectrometer. ^{1}H NMR spectra were recorded on a Bruker MSL-400 spectrometer (400 MHz). Chemical shifts were determined relative to TMS with residual signals of the corresponding solvents as internal standard.

Ethyl 2-Oxobutanoate (5a) was obtained by a method described in [20].

Ethyl 2-Oxohexanoate (5b). To a mixture of magnesium shavings (9.00 g, 370 mmol) in absolute THF (100 ml) a solution of 1-bromobutane (50.74 g, 370 mmol) in THF (50 ml) was added dropwise over 1 h with stirring. The reaction mixture was observed to become cloudy and the THF boiled. After addition of all the 1-bromobutane the reaction mixture was boiled for 10 min. Then, the freshly prepared solution of *n*-BuMgBr in THF was added dropwise with stirring over 0.5 h to a solution of diethyl oxalate (54.74 g, 375 mmol) in THF (50 ml) cooled to -50°C. The heating up occured of the reaction mixture, the temperature of which should not exceed -15°C. The reaction mixture was stirred for 2 h at -15°C and then a solution of 6 M HCl (60 ml) in 60 ml water was added. The organic layer was separated and washed with water (3×100 ml). The water layer was extracted with benzene (3×70 ml), the benzene layer was united with the organic layer, dried over $MgSO_4$, and the solvent removed in vacuum, to give a mixture (49.09 g) of diethyl oxalate and compound **5b**, with 54% of the latter according to ^{1}H NMR spectrum.

3-Ethyl-1,2-dihydro-2-oxoquinoxaline (6a) was obtained by the method described elsewhere [20].

3-**Butyl-1,2-dihydro-2-oxoquinoxaline (6b).** *o*-Phenylenediamine (30.86 g, 291 mmol) was added to a mixture (49 g) of diethyl oxalate and compound **5b** in 2-propanol (250 ml). The reaction mixture was stirred for 6 h at room temperature and kept overnight (after 5 min a precipitate was observed to form). The crystals of quinoxalinone **6b** were filtered off, washed with 2-propanol, and recrystallized. The filtrate was poured into water and kept overnight. The precipitated crystals of compound **6b** were filtered off, washed with water, and recrystallized.

1,3-Diethyl-1,2-dihydro-2-oxoquinoxaline (7) was prepared by a method described elsewhere [20].

3-Acetyl-1,2-dihydro-2-oxoquinoxaline (8a) was prepared by a method described elsewhere [20].

3-Butanoyl-1,2-dihydro-2-oxoquinoxaline (8b). A solution of chromic anhydride (1g, 10 mmol) in water (1 ml) and acetic acid (2 ml) were added with stirring to a solution of compound **7b** (1 g, 4.95 mmol) in acetic acid (17 ml) heated to 40°C. The mixture was stirred for 2 h at 80-85°C. The reaction mixture was cooled, poured into water, and extracted with chloroform (3×15 ml). The extract was washed with water (3×10 ml), the organic layer was died over $MgSO_4$, then passed through a column (300 x 15 mm) filled with 3g silica gel (L 100/160 μ), and washed with chloroform (80 ml). The solvent was removed at water pump vacuum to give analytically pure compound **8b**.

3-Acetyl-1-ethyl-1,2-dihydro-2-oxoquinoxaline (8c) was prepared by a method described elsewhere [20].

2-(2-Substituted benzimidazolyl)-3-alkyl-6- and -7-Substituted Quinoxalines 9, 13, 14 (General Method). The corresponding 1,2-phenylenediamine (0.55 mmol) was added to a solution of an alkanoylquinoxalin-2-one **8** (0.5 mmol) in acetic acid (5 ml), the solution was boiled for 1 h, cooled, and poured into water. The precipitated crystals were filtered off and washed with water.

1,5-Bis(3-ethyl-2-oxoquinoxalin-1-yl)-3-oxapentane (10). A mixture of 3-ethylquinoxalin-2-one (8.0 g, 45.98 mmol) and KOH (3.9 g, 68.4 mmol) in dioxane (200 ml) was heated to boiling, 1,5-dibromo-3-oxapentane (5.6 g, 24.10 mmol) was added, the mixture was boiled for 3 h, cooled, and poured into water. The crystals formed were filtered off, washed with KOH solution, then water, and recrystallized.

1,5-Bis(3-acetyl-2-oxoquinoxalin-1-yl)-3-oxapentane (11). CrO_3 (2.8 g, 28 mmol) in water (3 ml), and acetic acid (3 ml) was added with stirring to a solution of compound **10** (4.0 g, 9.53 mmol) in acetic acid (20 ml), the mixture was stirred for 1 h at 50-55°C and then for a further hour at 60-65°C. The mixture was poured into water, extracted with chloroform (5×20 ml), the organic layer was dried over magnesium sulfate, the filtrate was passed through a column filled with 3 g silica gel (L 100/160μ), washed with chloroform (100 ml) and the combined solution was evaporated in vacuum.

1,5-Bis[(3-methylquinoxalinyl-2)benzimidazol-2-yl]-3-oxapentane (12). *o*-Phenylenediamine (55 mg, 0.51 mmol) was added to a solution of compound **11** (100 mg, 0.22 mmol) in acetic acid (5 ml), boiled for 1 h, cooled, and poured into water. The precipitated crystals were filtered off and washed with water.

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